

Evaluating The Performance of Various Semiconductor Photocatalysts for Municipal Wastewater Treatment - Effects of Photocatalyst Type and Dosage

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Abstract—Water stress is a major concern as many cities worldwide face a rapidly depleting potable water supply. The prevailing water emergency requires a conscious effort to treat wastewater for reuse. The greatest challenge to accomplishing adequate wastewater remediation is maximizing the overall efficiency of wastewater treatment (WWT) systems. Advanced oxidation processes (AOPs) possess major prospects in WWT settings, if a suitable photocatalyst is considered. Therefore, this study aimed to evaluate the performance of various semiconductor photocatalysts for the treatment of municipal wastewater. The photocatalysts considered were Titanium dioxide (TiO_2), Iron III oxide (Fe_2O_3), Zinc Sulphate (ZnSO_4), and Copper Sulphate (CuSO_4). Also, two operating parameters such as catalyst load (0.5–2.5 g/L), and mixing speed (30–150 rpm) at constant UV-exposure time (45 mins) were investigated. To ascertain photocatalytic efficiency, the pH, colour, turbidity, and chemical oxygen demand (COD) of the treated effluent were monitored. At catalyst loading (1.5 g/ L), mixing speed (90 rpm), and UV-exposure time (45 minutes), CuSO_4 displayed the best results overall for COD removal efficiency of 72.47%, whilst ZnSO_4 was very efficient in removing turbidity and colour with removal efficiencies of 79% and 65.89% respectively. In this study, CuSO_4 was considered the most cost-effective (R 2.01) semiconductor photocatalyst to degrade the high organic content of wastewater.

Keywords—Advanced oxidation process, chemical oxygen demand, municipal wastewater, photocatalysts, semiconductors

I. INTRODUCTION

Water pollution and energy consumption that exceeds its supply, is a colossal threat because of globalization, urbanization, and anthropogenic activities [1].

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Wastewater brimming with toxic, non-biodegradable organic pollutants that are discharged into water bodies without adequate treatment poses severe risks, to human health, animal life, and can even obstruct the photosynthesis of plants, destroying the aquatic ecosystem [1-4]. Consequently, innovative, cost-effective, sustainable, and environmentally friendly water remediation techniques and materials are required to eliminate these dangerous threats.

Several approaches have been widely employed in industrial sectors for decontamination of wastewater, primarily including electro dialysis, membrane filtration, precipitation, adsorption, electrochemical reduction, and electrode ionization [5, 6]. However, these processes usually consume large amounts of energy and may be more complicated by transferring pollutants between different fluids, various wastes, and by-products generated by wastewater treatment plants [5]. Photocatalysis (advanced oxidation process) is recognized as a promising green solution in wastewater treatment settings to handle recalcitrant organic pollutants [5, 6]. The recent interest in the development of photocatalytic processes is based on the type of semiconductors with nontoxic effect, low cost, availability and convenient to use, high stability, and suitable band positions [7, 8].

Semiconductor photocatalysts have been proven successful candidates to convert toxic organic and inorganic water contaminant molecules into non-toxic molecules under the irradiation of light [9]. Dyes have received intense attention as model contaminants for photodegradation, including methylene blue (MB), methyl orange (MO), and rhodamine B (RhB) [10]. Photodegradation has also addressed antibiotics of growing concern such as tetracycline (TC), enrofloxacin (ENR), and other pharmaceuticals including diclofenac [6-10]. In addition, other toxic chemicals such as phenol and cyanide, heavy metal ions including Cr (VI), and bacteria like *E. coli* have been the subject of photocatalytic water treatment.

Remarkably studies reported stable molecules such as poly-fluoroalkyl substances (PFAS) were degradable with photocatalysis as well as recovering precious metals from e-waste (electronic waste) [5, 11-13]. This noteworthy reactivity towards various toxic substances makes semiconductor

photocatalysts a promising approach for sustainable environmental remediation and even resource recovery [10]. However, the current photocatalytic technology possesses several disadvantages that limit the widespread use of photocatalysts in practical industrial applications. The semiconductor photocatalysts prepared at this stage suffer from defects such as the large bandgap energy, the poor visible light absorption, and the ease of electron-hole / photo-generated charge carrier recombination (low quantum efficiency) that waste energy as heat, slow surface reaction kinetics, and low recyclability which result in poor photocatalytic activity and efficiency [14-18].

Consequently, industry demands, environmental and energy concerns prompted researchers to optimize the performance of the existing technology by acquiring highly efficient and visible light stable photocatalysts to improve photocatalytic efficiency and promote industrial photocatalytic technology, which is important for wastewater reclamation [19]. Moreover, the application of the photocatalysis process to treat real industrial wastewater requires a laboratory-scale feasibility study. Therefore, this study aimed to evaluate the performance of four semiconductor photocatalysts using a constant UV irradiation source for municipal wastewater treatment. The photocatalysts considered were Titanium dioxide (TiO₂), Iron (III) oxide (Fe₂O₃), Zinc Sulphate (ZnSO₄), and Copper Sulphate (CuSO₄). To ascertain, photocatalytic efficiency, water quality parameters including chemical oxygen demand (COD), turbidity, colour, and pH of the treated effluent were analysed to evaluate the effectiveness of the semiconductor photocatalysts at different catalyst loading/dosages. The respective semiconductor photocatalyst costs were then estimated and compared at the optimum catalyst load rate. The subsequent sections describe in detail the method (section II), results (section III), cost-benefits analysis (section IV), and the concluded findings (section V).

II. MATERIALS AND METHODS

A. Chemicals Used

All the chemicals and reagents used in this study were supplied by local South African suppliers are listed in Table 1.

TABLE I
CHEMICALS AND REAGENTS USED FOR THE STUDY

Photocatalyst Chemical Name	Chemical Formula	Supplier
Titanium (IV) Oxide	TiO ₂	Sigma Aldrich, Durban, South Africa and
<i>Iron (III) oxide / Ferric Oxide</i>	Fe ₂ O ₃	Sigma Aldrich, Durban, South Africa and
<i>Zinc Sulphate Heptahydrate A.R</i>	ZnSO ₄ •7H ₂ O	RADCHEM Laboratory supplies
<i>Cupric Sulphate Pentahydrate A.R</i>	CuSO ₄ •5H ₂ O	RADCHEM Laboratory supplies

B. Effluent Sample and Analytical Methods

Effluent sample

Synthetic wastewater was simulated using analytical-grade chemicals dissolved in 15 L of distilled water and 5 L of municipal wastewater. The composition of the chemicals used was adapted from Sibiya et al. [20]. The raw wastewater was collected from a local South African municipality wastewater treatment plant based in the Kwa-Zulu Natal province. The characterised raw wastewater sample was found to be of pH (6.95), colour (104 Pt.Co), turbidity (17.23 NTU), and COD (1716 mg/L). To maintain consistency of the effluent composition, further characterisation of the synthetic wastewater resulted in pH (6.14), colour (1812 Pt.Co), turbidity (506.3 NTU), and COD (1923 mg/L).

C. Analytical Methods

The pH and the turbidity were analysed using the pH meter HI98130 and the turbidity meter HI98703-02, respectively (HANNA instruments). The COD and colour were analysed by Spectrophotometer DR 3900 (HACH), using the stored programs 435- COD HR and 125- colour 465 nm. The COD, colour, and turbidity removal percentages were determined by using Eqs (1), (2), and (3), with the same equation set up applicable to turbidity percentage removal:

$$\text{COD removal \%} = \frac{C_i - C_f}{C_i} \times 100 \quad (1)$$

where C_i and C_f are the initial and the final COD concentrations (mg/L) before and after treatment, respectively [21].

$$\text{Colour removal \%} = \frac{Cl_i - Cl_f}{Cl_i} \times 100 \quad (2)$$

where Cl_i and Cl_f are the initial and the final Colour concentrations (Pt.Co) before and after treatment, respectively [22].

$$\text{Turbidity removal \%} = \frac{T_i - T_f}{T_i} \times 100 \quad (3)$$

where T_i and T_f are the initial and the final Turbidity concentrations (NTU) before and after treatment, respectively [22].

D. Experimental Setup

The experiment was carried out in a six-place jar testing apparatus (JLT 6, flocculation tester, Velp Scientific, New York, NY, USA). The setup consists of six identical beakers of 1 L, all equipped with a stirrer. Two 1-inch-diameter radiant fluorescent T8 black light blue bulbs, wavelength (400 nm), with a power rating of 18 W were used as the UV light source to excite the catalysts to trigger a reaction. The experiment varied catalyst load from 0.5–2.5 g/L in increments of 0.5 g/L, whilst exposure time and mixing speed remained constant at 45 mins and 90 rpm, respectively. The optimal catalyst load was then obtained and applied to the second experiment. The second experiment varied mixing speed from 30–150 rpm in increments of 30 rpm, whilst exposure time and catalytic load remained constant at 45 min

and 1.5 g/L respectively. The volume of effluent used for each beaker was 1 L. After each experimental run, the mixer was turned off, and the contents were allowed to settle for ~30 min and filtered using general laboratory filter paper and then collected in 50 mL sample bottles. Thereafter each photocatalyst was then analysed for pH, colour, turbidity, and COD removal.

III. RESULTS AND DISCUSSION

A. Effect of Catalyst Load on Photocatalysis

The photochemical impacts of the UV light used with a wavelength emitting span of 400 nm had a significant effect on each photocatalyst examined. Thus, the photocatalysis electron-hole generation was greatly influenced by the light intensity within the reactor, which influenced the pollutant transformation and destruction efficiency. By evaluating the catalyst load (0.5-2.5 g) of TiO₂, Fe₂O₃, ZnSO₄, and CuSO₄ in increasing sequence of 0.5g. It was observed that each catalyst load affected the photocatalytic degradation of the wastewater contaminants. As shown in Fig 1, the photocatalytic degradation efficiency first improved by the photocatalytic loading but decreased with excessive addition. Evidently, at the best catalyst load of 1.5g, the decreasing order of the COD removal (Fig 1a) by the catalyst was CuSO₄(73%) > Fe₂O₃(70%) > TiO₂(30%) > ZnSO₄(24%). This was due to the CuSO₄ with a bandgap of 2.2 eV (absorption wavelength 600 to 800 nm), had a good visible light absorption capacity, which produced electron holes to bond with the organic contaminants (COD). On the contrary, TiO₂ and ZnSO₄ with bandgaps of 3.2 eV and 3.6-3.8 eV respectively were difficult to oxidize or hydrolyze the organics at high electron-hole recombination rate [14]. In terms of turbidity removal (Fig 1b), at the same catalyst load of 1.5g, ZnSO₄ showed a good performance of 79% followed by Fe₂O₃(75%) > TiO₂(71%) > CuSO₄(61%). Moreover, the decolorization (Fig 2c) favored ZnSO₄(66%) > TiO₂(64%) > Fe₂O₃(57%) > CuSO₄(37%). It was deduced from the results that, the tendency of agglomeration such as particle-particle contact increased with respect to time, where active sites of the catalysts were ignited by the light source [23, 24].

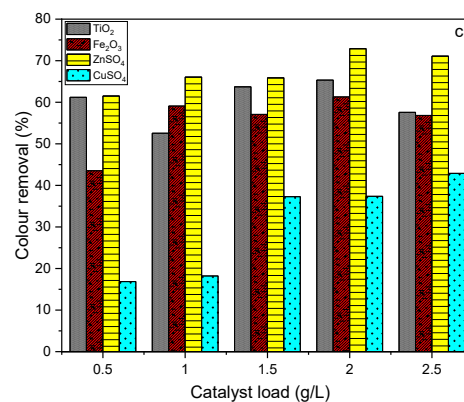
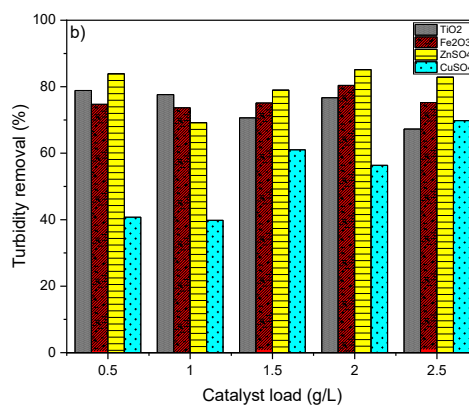
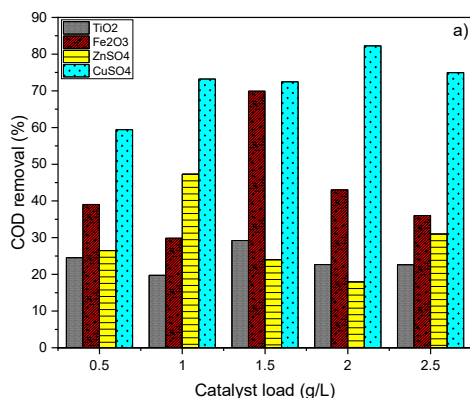


Fig. 1 Effect of catalyst load (0.5-2.5 g) of TiO₂, Fe₂O₃, ZnSO₄, and CuSO₄ for the removal of (a) COD, (b) turbidity, and (c) colour

B. Effect of Mixing Speed on Photocatalysis Treatment

The microstructure and homogeneity of the catalysts and the water molecules were influenced by the mixing speed. As shown in Fig 2, an increase in mixing speed (30-150 rpm) affected the catalyst-water molecules inter-particle reactions. The lower mixing speed (30 rpm) favored Fe₂O₃ > ZnSO₄ > CuSO₄ > TiO₂ for the removal of COD (Fig 2a). Fe₂O₃ semiconductor photocatalyst was the most effective for COD removal with a value of 68.35% at 30rpm. TiO₂ showed a COD removal efficiency of 65.96% at 60 rpm. ZnSO₄ and CuSO₄ resulted in COD removal efficiencies of 63.96% and 58.19% at 30 rpm respectively. This is because higher agitation introduces and increases the oxygenation of the water molecules which affects the amount of COD removed. There were oscillating trends observed with an increase in the mixing speed for the removal of both turbidity (Fig 2b) and colour (Fig 2c). In Fig 2b, CuSO₄ and ZnSO₄ semiconductor photocatalysts displayed the most efficacy for turbidity removal efficiency of 98.59% and 91.70%, at 90rpm and 120 rpm respectively. TiO₂ shows an optimum turbidity removal efficiency of 78.60% at 30 rpm. Fe₂O₃ displays an optimum turbidity removal efficiency of 74.39% at 120 rpm. In Fig 2c, Fe₂O₃, CuSO₄, and ZnSO₄ semiconductor photocatalysts displayed the most efficacy for colour removal efficiency at 51.31%, 92.17%, and 91.91%, at 90 rpm, and 120 rpm

respectively. Likewise, TiO_2 showed a colour removal efficiency of 55.98% at 30 rpm. This could be attributed to CuSO_4 and ZnSO_4 having higher stability, which require higher mixing speeds as compared to TiO_2 (powder) which tends to agglomerate in the water system, thereby increasing the turbidity and colour [23, 24]. Moreover, the high agitation develops more surface area contact interactions between particles of the catalyst and the water molecules. However, this affected the homogeneity of the water molecules and agglomeration of the particles [25]. This study conforms with the findings of previous work, which state that the degradation rate increases slightly with agitation speed [26, 27].

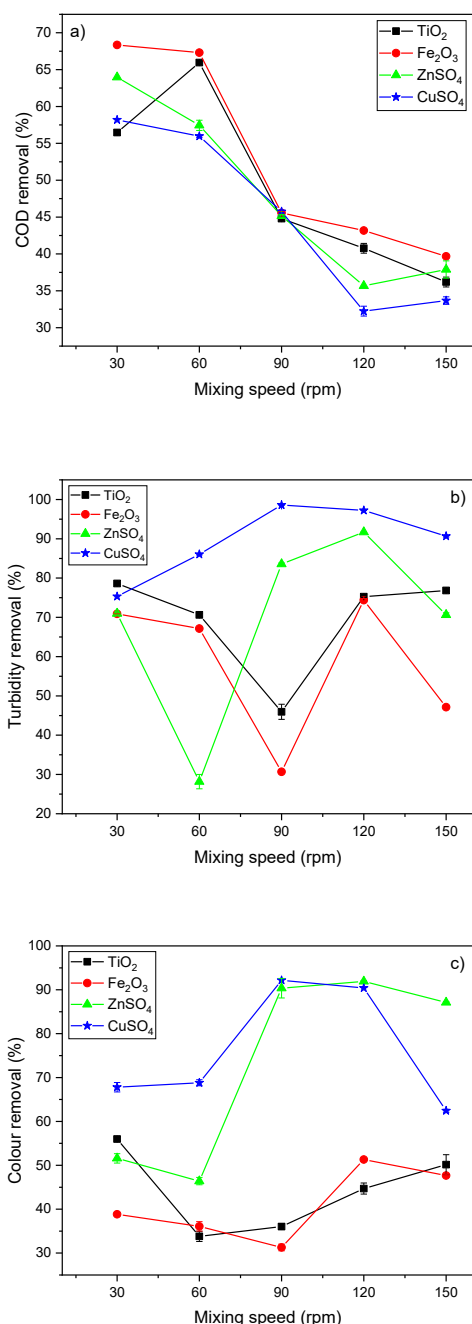


Fig. 2 Effect of mixing speed (30-150 rpm) on (a) COD, (b)

turbidity, and (c) colour removal using TiO_2 , Fe_2O_3 , ZnSO_4 , and CuSO_4

IV. COST BENEFIT ANALYSIS

The cost-benefit analysis of this study was estimated based on the cost of the catalyst at the desirable performed catalyst load (1.5g), which influenced the photocatalytic process [28]. Fig 3 shows the basic cost analysis of comparing the catalyst load at 1.5 g with the averaged desirability performance of removing the contaminants. CuSO_4 estimated cost at R2.01 was found to be the cheapest with $56,93 \pm 1.88\%$ treatability performance. Followed by ZnSO_4 with an estimated cost of R2.03 with $56,30 \pm 1.48\%$ performance. Likewise, Fe_2O_3 with an estimated cost of R5.21 and a treatment efficiency of $67,37 \pm 1.29\%$. Whereby TiO_2 had the highest cost (R32.47) with $54,52 \pm 1.82$ removal efficiency. Therefore, with desirable cost-effective performance at 50%, among the catalysts examined in this study, CuSO_4 was considered the best. This affirms that good water quality and maximum efficiency with the least quantity of catalyst load and cheaper cost are highly recommendable [29, 30]. CuSO_4 is superior and preferable due to it being economic viability and appropriate energy band (band gap of 2.2 eV), which has good visible light absorption capacity [31].

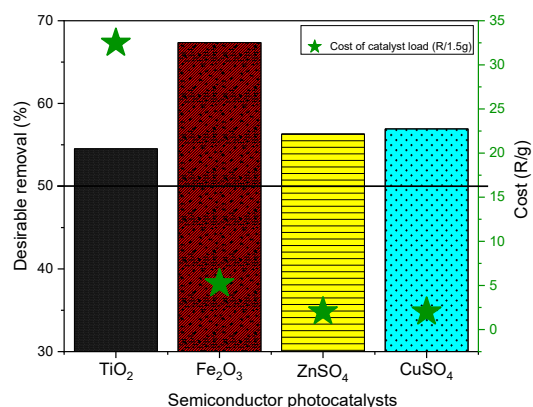


Fig. 3 Cost-benefit analysis of photocatalyst contaminant removal efficiency (%) at 1.5 g of catalyst load of TiO_2 , Fe_2O_3 , ZnSO_4 and CuSO_4

V. CONCLUSION

The degradation of high organic content of wastewater is very crucial, where the use of a photocatalytic process comes in handy. Herein, a cost-effective catalyst for the degradation of local South African wastewater was explored. Four semiconductor photocatalysts (TiO_2 , Fe_2O_3 , ZnSO_4 , and CuSO_4) were investigated to assess their treatability efficiency for the removal of COD, colour, and turbidity. Among the two operating factors investigated, catalyst load was found to have a significant impact on photocatalytic efficiency. Whereby, low catalyst load (1.5 g) and mixing speed (30 rpm) enhanced the aggregation and inter-particle surface contact time between the catalyst's active surface and water molecules.

Considering a desirability performance of reducing COD, turbidity, and colour by 50%, the increasing order of the estimated cost at 1.5g catalyst load was $\text{CuSO}_4(\text{R}2.01) < \text{ZnSO}_4(\text{R}2.03) < \text{Fe}_2\text{O}_3(\text{R}5.21) < \text{TiO}_2(\text{R}32.47)$. It was deduced that CuSO_4 was the cheapest alternative photocatalyst to the conventional TiO_2 , which has been the most widely used photocatalyst. Therefore, the prospect of CuSO_4 as a photocatalyst under an optimized photocatalytic process condition is viable for wastewater treatment.

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AUTHORS' CONTRIBUTIONS

Conceptualization, C.M., and E.K.T.; methodology, C.M.; software, C.M., and E.K.T.; validation, C.M., and E.K.T.; formal analysis, C.M., and E.K.T.; investigation, C.M.; resources, S.R. and E.K.T.; data curation, C.M.; writing—original draft preparation, C.M., and E.K.T.; writing—review and editing, C.M., S.R. and E.K.T.; visualization, C.M.; supervision, S.R., and E.K.T.; project administration, S.R. and E.K.T. All authors have read and agreed to the published version of the manuscript.

DECLARATION OF COMPETING INTERESTS

We have no financial or personal affiliations that could have influenced this paper's findings.

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